



LAWRENCE
LIVERMORE
NATIONAL
LABORATORY

Measure Of Quasi-Static Toughness And Fracture Parameters For Mock Explosive And Insensitive High Explosive LX-17

L. Ferranti Jr., F. J. Gagliardi, B. J. Cunningham,
K. S. Vandersall

March 11, 2010

The 14th International Detonation Symposium
Coeur d'Alene, ID, United States
April 11, 2010 through April 16, 2010

Disclaimer

This document was prepared as an account of work sponsored by an agency of the United States government. Neither the United States government nor Lawrence Livermore National Security, LLC, nor any of their employees makes any warranty, expressed or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States government or Lawrence Livermore National Security, LLC. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States government or Lawrence Livermore National Security, LLC, and shall not be used for advertising or product endorsement purposes.

Measure of Quasi-Static Toughness and Fracture Parameters for Mock Explosive and Insensitive High Explosive LX-17

*Louis Ferranti Jr., Franco J. Gagliardi, Bruce J. Cunningham and Kevin S. Vandersall

*Engineering Directorate, Engineering Technologies Division, Materials Engineering Group
Physical and Life Sciences Directorate, Chemical Sciences Division, Energetic Materials Program Element
Lawrence Livermore National Laboratory, Livermore, CA 94550

Abstract. The main motivation for this study is to develop an effective and simple method for preparing starter-cracks for fracture specimens of explosive materials. Evaluating the fracture behavior is an important aspect for safety and performance concerns in using these materials. The characterization of fracture behavior will enable the development and validation of computational models that can predict the behavior of damaged explosives. This method provides a consistent way to prepare accurate starter-cracks using a diamond wire saw and permits the fabrication of flexural bend fracture specimens from explosive material. With the advancement of consistent fracture specimens, toughness parameters such as critical strain energy release rate, G_{IC} and fracture toughness, K_{IC} were measured for mock explosive and the insensitive high explosive LX-17.

Introduction

Safety concerns and performance of damaged explosives have motivated significant experimental and modeling efforts to develop physically based material fracture models. Damage as a result of sufficient initial mechanical or thermal stimuli alters the material's combustion response, which may range from a mild reaction to a violent explosion. Identifying the thresholds for these reaction extremes and the evolution of material damage are needed to better predict the varying reaction behaviors resulting from the nature and level of initial stimuli.

A quantitative method for characterizing the fracture behavior is necessary for constructing and validating models that predict fracture initiation, evolution, and intensity of fragmentation or cumulative damage. Accurate fracture models are

essential for predicting the response to all varieties of stimuli. These include low level insults, ranging from accidental handling impacts, to more intense bullet and fragmented impacts, to low rate thermally induced damage. Obtaining direct measurements of toughness and fracture material parameters is desirable to quantify the mechanisms, evolution, and extent of damage.

In general, the compressive strength and the loading modulus of polymer bonded explosives (PBX) are moderately dependent on both strain rate and temperature.¹ Linear elastic fracture mechanics (LEFM) has classically been used to describe the behavior of sharp cracks in linear, perfectly elastic materials. But this method has also been successfully applied to non-linear materials that experience brittle failure.² The assumption of linear fracture behavior is particularly accurate for materials that have low

strength and fail in a brittle manner. Many polymers for example follow this response and have a very small plastic zone size ahead of the crack tip that makes the fracture process essentially linear elastic.² It is expected that this type of fracture behavior will also be consistent with many explosive materials that generally contain polymeric binders of 5-20 % by weight.³ Therefore, similar experimental techniques and analysis may also be applied successfully to energetic materials if they fail essentially in a linear elastic manner.

Fracture mechanics parameters such as fracture toughness, K_{IC} and critical strain energy release rate, G_{IC} characterize a material's intrinsic toughness and resistance to crack propagation for plane strain loading. The measurement of these parameters requires knowledge of specimen geometry and a pre-existing crack within the material. Controlling the uniformity and geometry of the starter-crack is the most critical factor for obtaining consistent results. Metallic materials have traditionally used fatigue pre-crack methods for growing a natural starter-crack. However, this process is very challenging for polymeric materials since fatigue crack growth is unstable, particularly for brittle materials. Many alternate techniques have typically been used for forming sharp starter-cracks within polymeric materials. These range from tapping, pressing, or sawing a razor blade into the specimen to slicing material with a surgical scalpel.⁴ A shortcoming of these techniques is the formation of a uniform and straight starter-crack without also causing an appreciable damage zone near the crack tip.

In the current study we consider the utility of a new notching technique, obtained from a precision diamond wire saw, for producing starter-cracks and evaluate its effectiveness for preparing fracture specimens for a mock explosive and an insensitive high explosive. The starter-cracks were nominally 300 μm thick with an equivalent diameter at the base or root of the notch. Quasi-static three-point bend fracture experiments were used to evaluate the starter-crack geometry using linear elastic fracture mechanics methods for computing toughness parameters. Initial tests were also conducted for poly(methyl methacrylate) (PMMA) specimens prepared using the diamond wire saw notching technique and compared to

traditional notching method measured values of G_{IC} and K_{IC} available in literature. The PMMA experiments provide a baseline for evaluating the sensitivity of the new notching technique and its use for measuring toughness parameters with explosive materials.

Experimental Details

Quasi-static fracture experiments were conducted on a specimen geometry consisting of a center-notched beam loaded in three-point bending. The experiment measures the energy dissipation associated with the growth of an already existing crack contained within an assumed linear elastic body that prescribes localized deformation at the crack tip through a linear load-deflection behavior. The analysis provides a geometrically independent measure of the toughness parameters by only varying the initial preferably sharp starter-crack length while keeping the specimen geometry constant.

For this analysis, the loaded beam of a sharply notched brittle material is assumed to deform in a totally elastic manner to the point of failure and the absorbed energy is sufficient to propagate the crack through the specimen.

Experimentally, G_{IC} can be obtained from the absorbed energy to failure, w for several starter-crack lengths using the following equation:

$$w = G_{IC} B D \phi \quad (1)$$

where B is the specimen thickness and D is the depth (see Figure 1), ϕ is the energy calibration factor which is a function of starter-crack length and may be calculated or measured experimentally.² Furthermore, K_{IC} can be obtained from the fracture stress, σ of a notched specimen using the following equation:

$$K_{IC} = Y \sigma \sqrt{a_0} \quad (2)$$

where a_0 is the notch or starter-crack depth and Y is a polynomial function of a_0/D which depends on the geometry and loading of the body.⁵ For both of these expressions, the measurements are obtained from a plane strain loading condition where crack instability occurs at a minimum value

of G_{IC} and K_{IC} . Additional details and the derivation of these expressions are described elsewhere.⁶

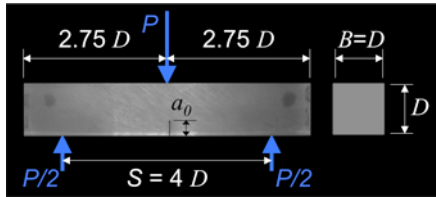


Figure 1. Single-edge notched bend (SENB) specimen geometry and load schematic shown with a PMMA specimen.

Materials

The materials studied in this investigation were a mock explosive (RM-03-AG) and an insensitive high explosive (LX-17-1). The mock explosive is an inert formulation used to replicate the density, as well as the mechanical and thermal properties of LX-17 and PBX 9502 explosives.⁷ It is also of interest to evaluate how well the mock explosive reproduces the toughness properties of the actual explosives.

The mock explosive contained 45 wt.% of cyanuric acid, 44.5 wt.% magnesium silicate, and 10.5 wt.% of poly(isobutyl-co-butyl methacrylate) (PIBBMA) binder. Several rectangular bar fracture specimens were machined from bulk parts obtained from die-pressed material using a 50.8 mm diameter steel die. The pressed parts had a right circular cylinder geometry approximately 63.5 mm tall with a similar diameter as the die. The mock explosive molding powder was pressed at 105 °C with an in-die pressure of approximately 207 MPa. Seven rectangular bar specimens were machined from each of the pressed parts. The measured specimen densities were 1.914 ± 0.003 g/cm³, 1.921 ± 0.004 g/cm³, and 1.919 ± 0.004 g/cm³ for the three prepared specimen batches.

The insensitive high explosive LX-17 was formulated with 92.5 wt.% TATB (triaminotrinitrobenzene) and 7.5 wt.% Kel-F 800 binder, a co-polymer of 75 wt.% chlorotrifluoroethylene and 25 wt.% vinylidene fluoride. The LX-17 molding powder was isostatically pressed at approximately 138 MPa and 105 °C. The final billet of material was

approximately 24.1 cm in diameter and 16.5 cm tall. Several rectangular bar specimens were machined from a single pressed billet. The average measured density obtained from thirty LX-17 fracture specimens was 1.894 ± 0.002 g/cm³.

Fracture specimens were also prepared from PMMA sheet material (Acrylite® GP Grade). These inert materials were initially used for establishing test procedures, specimen preparation techniques, and safety controls prior to experimenting with actual explosives. The PMMA specimens additionally provided a means for comparing traditional starter-crack methods thoroughly documented in the literature with those obtained from the precision diamond wire saw utilized in the current study.

SENB Specimen Preparation

Single-edge notched bend (SENB) specimens were machined into rectangular bar geometries from prepared bulk materials. All of the specimens were processed in an identical manner and had thickness and depth dimensions of 1.00 cm (B and D , respectively) and a length, L of 5.50 cm. A precision diamond wire saw was used to prepare various initial starter-crack lengths located at the mid-span length of the bar specimens. Figure 2 shows an example of an LX-17 SENB specimen exhibiting a representative starter-crack geometry ($a_0 = 4.028$ mm) for all the materials examined in this study. The wire saw produced smooth notches with a nominal kerf between 275 and 300 μ m depending on the material. Several starter-crack lengths were distributed evenly within a nominal range of 0.2 to 4.0 mm.

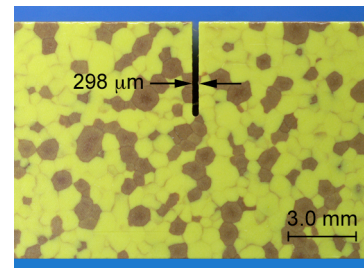


Figure 2. Image of an LX-17 specimen showing the typical starter-crack geometry (4.028 mm long with a 298 μ m kerf).

The precision wire saw, shown in Figure 3, employs a 220 μm diameter stainless steel wire with 40 μm grit diamonds imbedded into the surface. The wire is approximately 10 m long and wound onto a drum, which mounts onto a precision reciprocating motor with a continuously variable speed adjustment. Very little heat is generated during the cutting process because relatively fresh wire is constantly being presented to the cut, and heat is transferred to the brass drum that houses the wire. The cutting operation is performed remotely by advancing the wire saw with a low voltage step motor and an encoder for indicating the depth of cut. The small amounts of dust and debris generated during the cutting process are picked up by a filtered vacuum system.

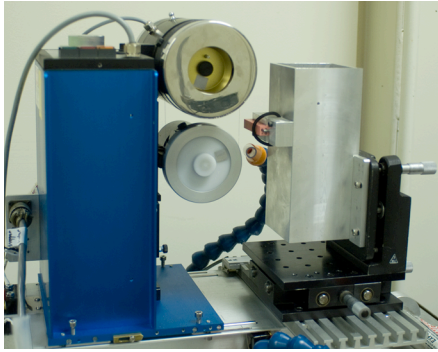


Figure 3. Image of the precision diamond wire saw used for cutting starter-cracks of various lengths into SENB specimens.

Fracture Toughness Test

Standard three-point flexural bend fracture experiments⁸ were performed on an MTS 858 mini-bionics II hydraulic load frame at ambient temperature ($\approx 21.5^\circ\text{C}$). The SENB specimens were loaded using displacement control at a constant speed of 2.54×10^{-3} mm/s. The specimen was positioned in a flexural fixture shown in Figure 4, which consisted of roller supports with a diameter of 9.525 mm fixed at a span, S of 40 mm (center-to-center). A special positioning fixture was designed to ensure the specimen was placed in the same position for each experiment with the starter-crack located at the exact center of the fixture span. The applied load, P load-point displacement, u and time, t were measured

throughout the experiment. The absorbed energy to failure, w for each flexural bend fracture experiment was obtained from integrating the applied load plotted as a function of load-point displacement.

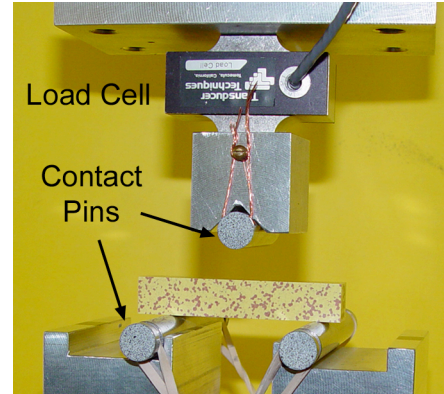


Figure 4. Image of the flexural (three-point) bend fixture used for measuring toughness parameters shown with an LX-17 specimen.

Results and Discussion

The quasi-static fracture toughness of mock explosive and LX-17 have been measured using flexural (three-point) bend experiments for specimens with a range of starter-crack lengths. The starter-cracks were fabricated using a precision diamond wire saw and had a kerf that was somewhat material dependent. The LX-17 starter-cracks had an average kerf of 295 ± 13 μm , while the mock explosive was slightly thinner with an average kerf of 273 ± 3 μm . It was apparent during fabrication that the LX-17 was softer and tended to leave material on the wire. This produced a slightly textured finish that showed a subtle waviness on the edges of the notch and produced a slightly wider kerf. The wire was conditioned using an alumina (Al_2O_3) block following each cut as a result. However, the mock explosive contained significant amounts of extremely hard magnesium silicate (44.5 wt.%), which provided better dimensional control of the notch and effectively self-conditioned the wire during the cut. The PMMA specimens showed a similar surface finish as the mock explosive, but did leave some material on the wire and require a conditioning step following each cut. The PMMA

starter-cracks had a comparable kerf to the mock explosive material with an average value of $276 \pm 6 \mu\text{m}$.

The fracture surfaces for both the LX-17 and the mock explosive are shown in Figure 5 for directly comparing the starter-crack surface finishes. The LX-17 specimen displays noticeable horizontal lines on the cut surface (top region of the specimen) while the mock explosive's finish is relatively smooth.

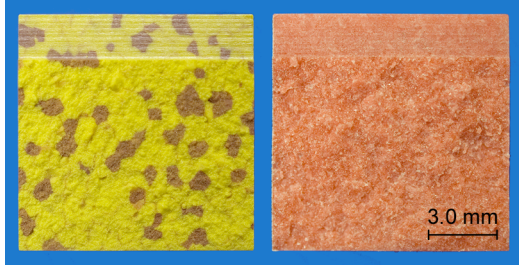


Figure 5. Images of LX-17 (*left*) and mock explosive (*right*) fracture surfaces. The starter-crack lengths are 1.934 mm and 1.827 mm for LX-17 and mock specimens, respectively.

The prepared starter-cracks for each of the materials exhibited excellent perpendicularity control and uniformity in length from one side of the specimen to the other as shown previously in Figure 2 for an LX-17 specimen. This is a significant benefit and provides very reliable starter-cracks for measuring consistent toughness parameters. Although every attempt was made to ensure the wire saw was perfectly aligned and perpendicular to the specimen face, there was a small amount of tilt associated with each cut. This corresponded to an average starter-crack difference of $37 \pm 13 \mu\text{m}$ from one side of the specimen to the other. The crack lengths were measured at six locations distributed evenly over the entire fracture surface following testing to avoid this influencing the experimentally measured toughness parameters. This proved to be the most reliable method for measuring the toughness parameters.

Quasis-static fracture experiments were performed for each of the materials at several starter-crack lengths. This included twenty-seven experiments for LX-17, fourteen for the mock explosive, and twenty-two for PMMA. Representative load-displacement curves are

shown for LX-17 and the mock explosive in Figure 6 and Figure 7, respectively. All of the curves show a characteristic brittle response with a linear rise with the peak load corresponding to the point of fracture. This is also evident from the fracture surfaces shown in Figure 5. The PMMA specimens demonstrated a similar response and brittle fracture surfaces. The load magnitude is inversely proportional to the initial starter-crack length, and the area under the curve represents the stored or absorbed elastic energy. The longer starter-cracks display a slightly different loading slope and may possibly exhibit some plasticity. This appears to be evident for starter-cracks of approximately 2 mm for both LX-17 and the mock explosive and gradually becomes more apparent as the starter-crack length increases. However, this did not appear to influence the overall measured toughness parameters.

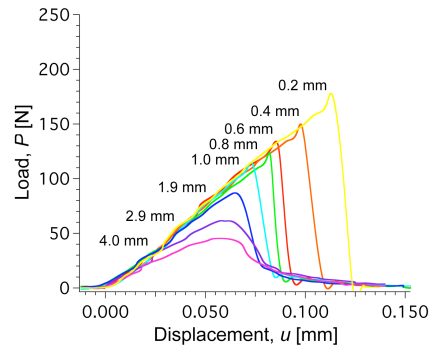


Figure 6. Representative load-displacement data for LX-17 specimens with several starter-crack lengths ($0.2 \text{ mm} \leq a_0 \leq 4.0 \text{ mm}$).

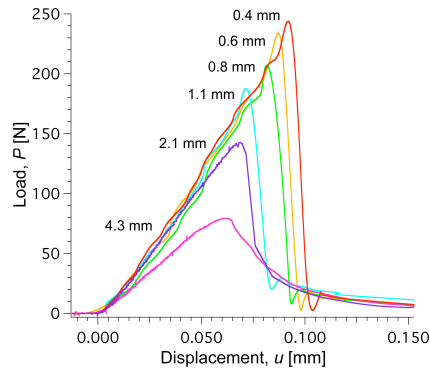


Figure 7. Representative load-displacement data for mock explosive specimens with several starter-crack lengths ($0.4 \text{ mm} \leq a_0 \leq 4.3 \text{ mm}$).

The specimens typically fractured uniformly throughout the thickness in a fairly straight direction away from the starter-crack. The fracture initiation site was typically in the center of the starter-crack root diameter. However, some experiments showed the tendency for the fracture initiation site to be closer to the edge of the starter-crack. An example of this is shown in Figure 8 for an LX-17 specimen with a starter-crack length of 0.632 mm. This particular specimen also shows an example of the crack path following the prill boundary as indicated by the arrow in the figure. This response was exhibited by a small fraction of the specimens examined in this study.

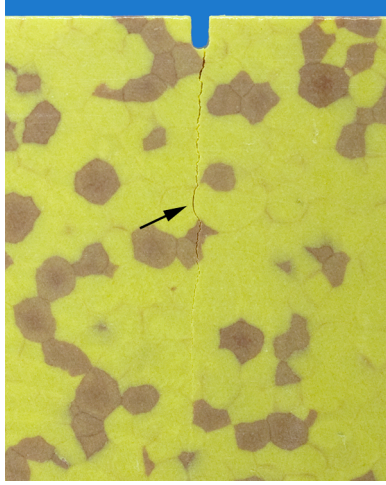


Figure 8. Post-fracture LX-17 SENB specimen image showing a crack initiating from the root (off-center) of a 0.632 mm long starter-crack. The arrow indicates a location where the fracture path follows the contour of a prill.

The absorbed elastic energy, w during fracture was obtained by integrating the load-displacement curves for each flexural bend experiment. The absorbed energy was plotted as a function of the specimen geometry given by $BD\phi$. The results for LX-17 and the mock explosive are shown together in Figure 9. The critical strain energy release rate, G_{IC} was obtained from the slope according to equation (1). These values are listed in Table 1 for LX-17, the mock explosive, and PMMA. Both the LX-17 and mock explosive exhibit two different slopes above a critical energy of approximately $0.6 \text{ J} \times 10^{-2}$. These correspond to starter-crack lengths

of 0.4 to 0.8 mm for the mock explosive and 0.2 to 0.4 mm for LX-17. It is currently unclear why there is a G_{IC} transition above this absorbed fracture energy. However, it may be possible that the shorter starter-crack lengths change the character of the fracture mechanism by introducing microcracking or ductile yielding or a combination of both. It is also possible that the crack speed may increase for these shorter starter-cracks and changes the strain rate at the crack tip. Marshall, *et al.* have shown crack speed can have a considerable effect on toughness of PMMA, because the modulus and yield stress are sensitive to changes in strain rate.⁹ This result requires further detailed study and analysis of the fracture surfaces.

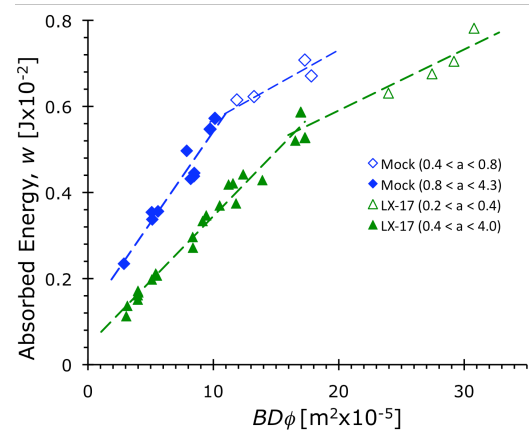


Figure 9. Results obtained from flexural bend experiments for mock explosive and LX-17 specimens with several starter-crack lengths. Plot shows the absorbed energy, w as a function of specimen geometry $BD\phi$ for calculating G_{IC} from the slopes.

Table 1. Experimentally measured toughness values K_{IC} and G_{IC} over a range of starter-crack lengths for mock explosive, LX-17, and PMMA.

	a_0 Range [mm]	$BD\phi$ Range [$\text{m}^2 \times 10^{-5}$]	K_{IC} [$\text{MPa}\sqrt{\text{m}}$]	G_{IC} [kJ/m^2]
Mock	0.8 – 0.4	10.2 – 18.3	0.602 ± 0.030	0.016
	4.3 – 0.8	2.9 – 10.2	0.659 ± 0.026	0.042
LX-17	0.4 – 0.2	18.3 – 34.6	0.317 ± 0.024	0.012
	4.0 – 0.2	3.1 – 18.3	0.366 ± 0.019	0.030
PMMA	3.7 – 0.3	3.3 – 23.7	1.749 ± 0.325	1.157

The fracture toughness, K_{IC} was also determined for each material from the peak load and equation (2). The maximum stress for a flexural bend test is given by:

$$\sigma = \frac{P_{\max}}{BD} \quad (3)$$

These values are also listed in Table 1 for each of the materials studied in this work.

The magnitudes for both G_{IC} and K_{IC} for the mock explosive was slightly greater than that of LX-17 over the entire range of starter-crack lengths examined. This result seems appropriate since the peak loading level at which fracture took place for the mock explosive was greater. The measured toughness behavior from the current study shows that the mock explosive exhibits similar properties to LX-17. This is in accordance with previous studies which have shown LX-17 and this mock explosive to have similar mechanical properties, such as tensile strength.¹⁰

Flexural bend experiments were conducted to evaluate the effectiveness of starter-cracks fabricated using a diamond wire saw. The toughness properties of PMMA have been extensively studied and were used in this work to evaluate the effectiveness of the starter-crack fabrication method. Although the starter-cracks are significantly thicker than a natural crack and have a blunt tip, the experimentally measured toughness value of $1.749 \pm 0.325 \text{ MPa}\sqrt{\text{m}}$ compares well with those available in literature. Marshall and Williams have summarized many of the published results for the fracture toughness of PMMA. These values range 0.93 to $1.75 \text{ MPa}\sqrt{\text{m}}$ for flexural bend fracture experiments using SENB specimens.¹¹ Hashemi and Williams measured a consistent value of $1.8 \text{ MPa}\sqrt{\text{m}}$ while varying the specimen thickness.¹² The critical strain energy release rate was 1.157 kJ/m^2 for PMMA, slightly lower than the published value of 1.28 kJ/m^2 .²

Summary and Conclusion

The critical strain energy release rate, G_{IC} and fracture toughness, K_{IC} were measured in this study for a mock explosive and the insensitive high explosive LX-17. The flexural (three-point)

bend experiments used an SENB specimen with a starter-crack located at the mid-span length of the specimen. The starter-cracks were fabricated using a precision diamond wire saw. The wire saw produced starter-cracks at various lengths between 0.2 and 4.0 mm with a nominal kerf of $300 \mu\text{m}$. The fabrication method provided a highly repeatable geometry that presented meaningful fracture toughness parameters, although these starter-cracks were significantly thicker than a natural crack. The blunt notch did not appear to influence the measured toughness parameters for PMMA, which were used to calibrate the sensitivity of this technique.

The load-displacement plots showed a characteristic brittle response for both the mock explosive and LX-17. The fracture response was mostly elastic in nature with only minimal non-linear behavior. This permitted the calculation of toughness parameters using linear elastic fracture mechanics methods.

Future work will examine the influence of the starter-crack tip geometry by conducting an additional processing step to sharpen the root of the notch. This will provide a direct way to measure the toughness parameters with a geometry that is closer to a natural crack. Additional dynamic fracture experiments, utilizing the same specimen geometry evaluated in the current study, will be performed to measure strain rate effects for these materials. It is also possible to transition the techniques and methods of this study to other explosive formulations and propellants.

Acknowledgements

The authors would like to thank Frank Garcia, Dan Greenwood, Greg Silva, and Jeff Wardell for their technical support and assistance in this study. This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

References

1. Blumenthal, W.R., Gray III, G.T., Idar, D.J., Homes, M.D., Scott, P.D., Cady, C.M. and Cannon, D.D., "Influence of Temperature and Strain Rate on the Mechanical Behavior of PBX

- 9502 and Kel-F 800,” *Shock Compression of Condensed Matter* – 1999, **Vol. 505**, pp. 671-674 (2000).
2. Plati, E. and Williams, J.G., “The Determination of the Fracture Parameters for Polymers in Impact,” *Polymer Engineering and Science*, **15**, [6], pp. 470-477 (1975).
 3. Cooper, P.W. and Kurowski, S.R., “Introduction to the Technology of Explosives,” New York: Wiley-VCH, pp. 21-23 (1996).
 4. Lu, X., Qian, R. and Brown, N., “Notchology – The Effect of the Notching Method on the Slow Crack Growth Failure in a Tough Polyethylene,” *Journal of Materials Science*, **26**, pp. 881-888 (1991).
 5. Brown, Jr., W.F. and Srawley, J.E., “Plane Strain Crack Toughness Testing of High Strength Metallic Materials,” ASTM STP 410 (1966).
 6. Williams, J.G., “Introduction to Linear Elastic Fracture Mechanics,” in Fracture Mechanics Testing Methods for Polymers, Adhesives, and Composites, eds. Moore, D.R., Pavan, A., and Williams, J.G., pp. 3-26 (2001).
 7. Hoffman, D.M., Cunningham, B.J., and Tran, T.D., “Mechanical Mocks for Insensitive High Explosives,” *Journal of Energetic Materials*, **21**, [4], pp. 201-222 (2003).
 8. “Standard Test Method for Plane-Strain Fracture Toughness and Strain Energy Release Rate of Plastic Materials,” ASTM D 5045-99 (2007).
 9. Marshall, G.P. and Williams, J.G., “The Correlation of Fracture Data for PMMA,” *Journal of Materials Science Letters*, **8**, pp. 138-140 (1973).
 10. Groves, S. and Cunningham, B.J., “Tensile and Compressive Properties of Billet Pressed LX17-1 As a Function of Temperature and Strain Rate,” LLNL Report UCRL-ID-137477 (2000).
 11. Marshall, G.P., Culver, L.E., and Williams, J.G., “Crack and craze propagation in polymers: A fracture mechanics approach,” *Plastics and Polymers*, **37**, [1], pp. 75-81 (1969).
 12. Hashemi, S. and Williams, J.G., “Size and Loading Mode Effects in Fracture Toughness Testing of Polymers,” *Journal of Materials Science*, **19**, pp. 3746-3759 (1984).